

Ion induced weight loss and thermal gravimetric analysis of ion-irradiated polyvinyl formal

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Polyvinyl polymers are high-performance insulating materials used in many applications. At the new *Facility for Antiproton and Ion Research* (FAIR) polyvinyl polymers are foreseen as wire insulation in some parts of the superconducting magnets [1]. During the operation of FAIR, beam losses will result in secondary radiation fields consisting of various energetic particles and gamma radiation. Ion-induced radiolysis of polymers involves bond scission, cross-linking and other chemical reactions leading to outgassing of small volatile degradation products and finally to loss of functional properties. At operational cryogenic temperatures, these gases may accumulate and raise problems during thermal cycling of the superconducting magnets [2]. This study extends existing data of beam-induced weight loss in polymers and shows results of thermal gravimetric analysis (TGA) of irradiated polyvinyl formal. Results are complementary to previous reports on infrared spectroscopy (IR) and qualitative interpretation of residual gas analysis [3].

20- μm thick Formvar® foils were synthesized and irradiated at the X0 beamline of the UNILAC with Au and U ions of 11.1 MeV/u. The applied fluences were 1×10^9 - 6×10^{11} Au/cm² for TGA measurements and 1×10^{10} - 1×10^{12} U/cm² for weight-loss measurements. TGA was performed using a Netzsch TG209 F1 instrument with a heating rate of 4 K/min in argon atmosphere. For weight loss analysis a chemical balance with a resolution of about 0.1 mg was used.

TGA measures the weight of a material as a function of temperature in a controlled atmosphere. Figure 1 compares the decomposition behavior of a pristine and an ion-irradiated polyvinyl formal sample from room temperature to 650 °C. The pristine Formvar® foil shows two steps, first a small initial weight loss occurring between 104 and 153°C which is commonly attributed to the release of water absorbed in the polymer. Between ~300 and 420°C a significant weight loss occurs with a 5% of char residue. The decomposition of nearly the entire polymer indicates that the thermal degradation follows a depolymerisation process and not a so-called random scission process, which would lead to a great amount of char. The TGA curve of the irradiated sample shows only one broad degradation phase occurring between 110 and 420°C with a residual char of 10%. The temperatures for maximum mass loss rate ($T_{d(max)}$) of both samples are indicated in the derivative curves (Fig. 1, bottom). The ion irradiation leads to a slight increase of $T_{d(max)}$. Both effects are explained by ion induced synthesis of carbon clusters which are not decomposed at the used temperatures.

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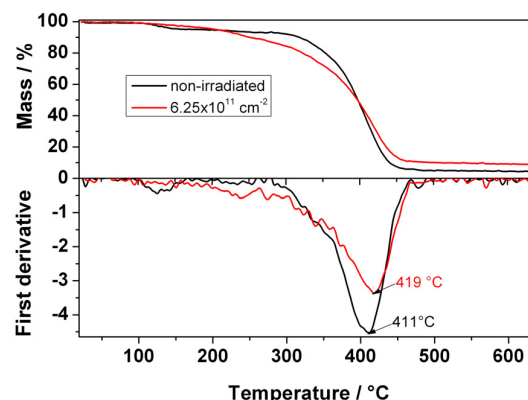


Figure 1: TGA curves of Formvar® foils (black) before and (red) after irradiation with 6×10^{11} Au-ions/cm² (top) and their derivatives (bottom).

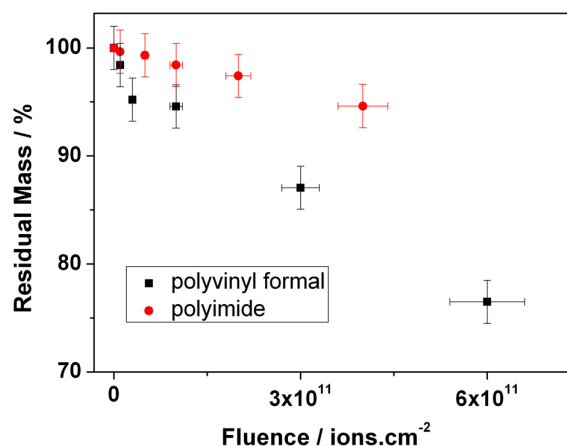


Figure 2: Residual mass as a function of ion fluence for polyvinyl formal (black) and polyimide (red) [2] irradiated with 11.1-MeV/u U ions.

Figure 2 depicts the residual mass of irradiated polyvinyl formal in comparison with results from irradiated polyimide samples from previous work [2]. All irradiated samples show a strong decrease in weight with increasing ion fluence. Compared with polyimide a greater decrease is found for polyvinyl formal. Polyimide insulation obviously forms less outgassing radiolysis products which could accumulate when used in cryogenic environment.

[1] T. Seidl et al. Nucl. Instr. Meth. B 272, (2012), 400.

[2] D. Severin, PhD thesis, Univ. Marburg (2008).

[3] U.H. Hossain et. al., GSI Scientific Report; PNI-MR-20 (2011).